

## PATENT ABSTRACTS OF JAPAN

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## (54) PRODUCTION OF GLASS SUBSTRATE FOR MAGNETIC RECORDING MEDIUM

## (57)Abstract:

PROBLEM TO BE SOLVED: To attain enhanced work efficiency, a reduced cost and high productivity in the production of a glass substrate for an information recording medium by simultaneously carrying out chemical tempering and alkali removal treatment.

SOLUTION: When a glass substrate is chemically tempered with a tempering salt bath, the nitrate of a metal having a smaller ionic radius than ions in the tempering salt bath, e.g. silver nitrate is added to the tempering salt bath by 0.1-10 wt.% and the chemical tempering and alkali removal treatment of the glass substrate are carried out by one-step treatment using the resulting salt bath. The chemical tempering and alkali removal treatment can be continuously and simultaneously carried out.

## LEGAL STATUS

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**CLAIMS**

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[Claim(s)]

[Claim 1] The glass substrate manufacture approach for information record media characterized by performing dealkalization processing to coincidence with a chemical strengthening by adding a metaled nitrate to a strengthening salt bath, and a substrate being immersed in this strengthening salt bath in chemical-strengthening processing of the glass substrate for information record media in order [ which is depended on the ion exchange ] to carry out chemical-strengthening processing.

[Claim 2] The glass substrate for information record media manufactured in chemical-strengthening processing of the glass substrate for information record media by performing dealkalization processing to coincidence with a chemical strengthening by adding a metaled nitrate to a strengthening salt bath, and a substrate being immersed in this strengthening salt bath in order [ which is depended on the ion exchange ] to carry out chemical-strengthening processing.

[Claim 3] Strengthening, the dealkalization salt bath which added the nitrate of the metal which is larger than the ionic radius of the alkali ion in glass, and has an ionic radius smaller than the ionic radius of each ion in a strengthening salt bath especially in the manufacture approach of the glass substrate for information record media of claim 1 0.1 to 10% of the weight in the strengthening salt bath.

[Claim 4] Strengthening of claim 3, the glass substrate for information record media manufactured using a dealkalization salt bath.

[Claim 5] Strengthening, the dealkalization salt bath which added especially the silver nitrate in strengthening of claim 3, and a dealkalization salt bath.

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**DETAILED DESCRIPTION**

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the information record medium which used this glass substrate for the manufacture approach of the glass substrate for information record media, and the list.

[0002]

[Description of the Prior Art] Although the substrate for information record media has been conventionally offered by the substrate made from aluminum, the substrate made from carbon, a polycarbonate substrate, etc., since surface smooth nature is excellent, the glass substrate attracts attention with the formation of amount-of-information increase in recent years. Since the surface smooth nature of a glass substrate does not have a grain boundary in glass by development of a polish technique, it is possible to reduce surface roughness to atomic level theoretically. Moreover, by the fall of the display flatness of a disk substrate, it became possible to reduce the surfacing height of the magnetic head for reading, and the recording density per unit area went up, and has led to the increment in storage capacity. Thus, although the glass disk substrate attracts attention, there is a fault of being easy to damage with the impact at the time of the handling in the production process of the glass for substrates etc., and strengthening processing by the ion exchange is usually performed.

[0003] However, it poses a problem that the rich layer of the alkali metal of a strengthening layer especially a potassium atom, or a sodium atom appears on a disk main front face in the substrate front face after chemical-strengthening processing of a glass substrate here. There is a danger that the migration of alkali-metal ion will cause reading incorrect actuation from this layer as the state of preservation of a disk and aging after subsequent information record-medium membrane formation because the carbonate and chloride of alkali metal deposit on a disk edge and the main front face. Moreover, alkali metal reacting with the record medium of a metal alloy, and causing incorrect actuation of equipment is also considered.

[0004] For this reason, after carrying out ion exchange treatment of the glass substrate front face, the alkali-metal ion of the maximum surface layer which is easy to be eluted is removed, and the dealkalization processing for raising chemical resistance and the cure against the closure of alkali-metal ion are performed. For example, there is an approach using an acid, especially strong acid like the approach of making the heated concentrated sulfuric acid contacting like the manufacture approach shown in a patent public presentation number and JP,10-226539,A. Moreover, like the manufacture approach shown in a patent public presentation number and JP,8-180402,A, into 80-100-degree C warm water, there is also a warm water art which carries out dealkalization metal ion processing of the glass substrate after ion exchange treatment by carrying out immersion processing for about 2 to 10 hours, in this case, impregnation processing of a divalent metallic ion is carried out to the maximum surface layer of the glass substrate after dealkalization metal ion processing, and the cure against the closure of alkali-metal ion is added further.

[0005]

[Problem(s) to be Solved by the Invention] However, since [ which needs immersion time amount at least 2 hours or more ] there are few treatment effects, impregnation processing of a divalent metallic ion is also required of the danger on the handling of using strong acid, such as heat concentrated sulfuric acid (it being heat concentrated sulfuric acid of 96% or more of concentration for example, at 100-degree-C \*\*), in these dealkalization metal ion processings, and warm water processing as after treatment. moreover, the process of [ in order for the dealkalization processing currently performed from these former to perform dealkalization processing after chemical-strengthening processing, to be carried out at a chemical strengthening and two processes of dealkalization and to avoid the drag-in to the dealkalization processing tub of a strengthening salt further ] strengthening, washing, and dealkalization processing — not constructing — it does not obtain but has also become a cost rise.

[0006] This invention makes it the technical problem to offer the glass substrate for information record media created by the effectiveness rise of workability, low-cost-izing, the chemical-strengthening art of the glass substrate for information record media which realizes the sex from Takao and a dealkalization art, and its approach by performing chemical-strengthening processing and dealkalization processing to coincidence in the manufacture approach of the glass substrate for information record media.

[0007]

[Means for Solving the Problem] this invention person is related with the manufacture approach of the glass substrate for information record media, as a result of examining the above-mentioned technical problem wholeheartedly. The nitrate of the metal which has a bigger ionic radius smaller than potassium ion than sodium ion in strengthening salt baths, such as mixed salt of the potassium nitrate or potassium nitrate used for the chemical strengthening from the former, and a sodium nitrate, is added. By performing chemical-strengthening processing and dealkalization processing of a glass substrate to coincidence, a header and this invention were completed for the above-mentioned technical problem being attained by performing the same strengthening processing as usual.

[0008] That is, the glass substrate for information record media of this invention is carrying out predetermined time immersion of the non-strengthened glass substrate at predetermined temperature at the salt bath for chemical strengthenings, and is the creation approach of the new glass substrate for information record media of performing dealkalization to a glass substrate by one processing within the same processing tub with installation of a chemical-strengthening layer.

[0009]

[Embodiment of the Invention] Hereafter, this invention is explained to a detail.

[0010] this invention person sets to chemical-strengthening processing of a glass substrate especially paying attention to strengthening processing and dealkalization processing of the glass substrate for information record media. It is characterized by

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performing chemical-strengthening processing and dealkalization processing of a glass substrate by one processing within the same processing tub by adding a metal nitrate further from the former to a strengthening salt. By adding the nitrate of several% of the weight of a metal to the weight of the salt bath for strengthening, a header and this invention can be made for it being possible to perform chemical-strengthening processing and dealkalization processing to coincidence continuously.

[0011] In chemical-strengthening processing of the glass substrate for information record media, the alkali ion exchange on the front face of glass is performed at the temperature below a glass transition point, and, as for the case of one-step strengthening, in exchange of the sodium ion in glass, and the potassium ion in a strengthening salt bath, and two-step strengthening, exchange of the lithium ion in glass, sodium ion and the sodium ion in a strengthening salt bath, and potassium ion is performed. The thermal diffusion speed of the alkali-metal ion in this case is in inverse proportion to the ionic radius of each ion. Generally, under this temperature, this \*\*, and this concentration, the direction of ion with a small ionic radius has high potential energy, and can say that activity is high. Therefore, by adding the nitrate of a metal with the middle ionic radius of the ionic radius of exchangeable ion in a chemical-strengthening salt bath, the alkali ion with the small ionic radius in glass is emitted by thermal diffusion into a salt bath from a glass front face, and controls the rapid ion exchange on the front face of glass of the alkali ion which has a big ionic radius in a salt bath further. That is, the amount of alkali metal with the small ionic radius which is easy to separate after glass strengthening is decreased, and the chemical-strengthening processing by the ion exchange is attained at coincidence. That is, it is related with the glass substrate for information record media manufactured by the manufacturing method of the glass substrate for information record media using the strengthening salt bath which made it possible to perform the strengthening processing and dealkalization processing by the ion exchange on the front face of glass to coincidence, and enabled the productivity rise and the cost cut, and its strengthening salt bath, and its approach.

[0012] Moreover, by applying this invention, the productivity drive of the glass substrate for information record media and cost cut-ization can be attained. It cannot limit especially as a glass ingredient and for example, aluminosilicate system glass, soda lime glass, borosilicate glass, alumino borosilicate glass, etc. can be used as what can form a strengthening layer by chemical-strengthening processing. Moreover, limitation does not have the surface roughness of the glass substrate before performing strengthening processing, either.

[0013] However, it is required that the chemical-strengthening processing in the temperature below the glass transition point of tempered glass-ed is possible and for the nitrate of the metal added in the temperature to dissolve into a strengthening salt bath. Moreover, it is convenient, even if it is desirable that the ionic radius in the inside of the salt bath of this nitrate metal to add is within the middle of the ionic radius in each ionic state of the alkali metal in glass and a salt bath Nakakane group and it becomes large from it. In addition, 0.1 or less % of the weight of the dealkalization effectiveness is insufficient, 10% of the weight or more of the amount of dealkalization is large, and glass colors it yellow with complex ion, and is also a cost rise, and the amount which adds a metal nitrate in a strengthening salt bath does not have it. [ desirable ]

[0014]

[Example] (Example 1) The salt bath which added the 30 sections for the sodium nitrate and added the 1 section of silver nitrates for the potassium nitrate during the mixed salt bath of the 70 sections was made into the chemical-strengthening salt bath. This glass substrate ingredient was taken out from the chemical-strengthening salt bath tub after 40 minutes - 6-hour immersion using the alumino silicate glass which carried out the disk configuration whose surface roughness Ra is 0.3 micrometers, and whose diameters are 95mmphi and the thickness of 1.07mm as a glass substrate ingredient for [ strengthened ] in the chemical-strengthening salt bath maintained at the range of 360 degrees C - 450 degrees C, and the deposit salt adhering to this glass substrate after cooling was washed out with water. This was made into the strengthening sample. About the depth of the chemical-strengthening layer formed in the glass front face, it ground to the flake so that this sample could be measured with a polarization microscope, and polarization microscope observation was performed. The result was shown in Table 1.

[0015]

表 1. 処理温度と時間の変化による強化層厚みの変化

単位:  $\mu\text{m}$  (片面)

		処 理 時 間			
		40分	80分	160分	240分
処 理 時 間	360℃	30	30	40	40
	380℃	40	50	60	60
	400℃	70	90	100	100
	450℃	80	90	110	110

As shown in Table 1, it turns out that a strengthening layer (compressive-stress layer) can be introduced into a glass front face by using this invention, and the breakage in the disk breakage at the time of handling, a subsequent polish process, etc. can be mitigated sharply.

[0016] And among each sample shown in Table 1, the waterproof trial was performed about the sample processed for 40 minutes, and 380 degrees C of measurement of the alkali-metal (Na, Li, K) concentration eluted from glass were measured with atomic-absorption-analysis equipment. In addition, the waterproof trial was immersed into the Teflon (trademark) beaker which filled the glass substrate with 50ml distilled water, moved the Teflon beaker containing the glass substrate to the constant temperature bath kept at 80 degrees C, and carried out quantitative analysis of the amount of alkali metal which held for 24 hours and was eluted from the glass substrate in 80-degree C warm water with the atomic absorption method. The result was as being shown in Table 2.

[0017]

表 2. ガラスディスクのアルカリ溶出量

単位:  $\mu\text{g}/\text{cm}^2$

	Na	Li	K	合 計
ブランク	0.35	0.125	0	0.475
比較例 1	0.315	0.007	0.16	0.482
実施例 1	0.15	0.007	0.05	0.207

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- A blank is the processing [ in which it does not strengthen ] article of alumino silicate glass.

The example 1 of a comparison is alumino silicate glass which carried out strengthening processing with the strengthening salt bath which mixed the potassium-nitrate 70 section and the sodium-nitrate 30 section.

- An example 1 is alumino silicate glass processed by this invention approach.

It turns out that the alkali elution volume from glass is decreasing or less [ those ] to 1/2, and chemical resistance is improving very much compared with that in which the glass substrate manufactured by the approach of this invention carried out strengthening processing with non-strengthened elegance or the usual chemical-strengthening salt bath so that clearly from Table 2.

[0018] (Example 2) Next, the salt bath which added the 1 section of silver nitrates for the potassium nitrate during the mixed salt bath of the 100 sections was made into the chemical-strengthening salt bath. About the sample which carried out chemical-strengthening processing of the soda lime glass of the same configuration as an example 1 similarly, the strengthening layer depth and an alkali elution volume were measured by this approach. Consequently, among each sample shown about the depth of a strengthening layer in Table 3 and 3, the waterproof trial was performed about the sample processed for 16 hours, and 450 degrees C of results of having measured the alkali elution volume from glass were shown in Table 4.

[0019]

表 3. 処理温度と時間の変化による強化層厚みの変化

単位:  $\mu\text{m}$  (片面)

		処 理 時 間		
		1 時間	4 時間	1 6 時間
処 理 時 間	4 0 0 °C	1 0	2 5	5 0
	4 5 0 °C	1 0	2 5	6 0
	4 8 0 °C	1 5	3 0	7 0

As shown in Table 3, it turned out that this invention can introduce a strengthening layer (compressive-stress layer) into a glass front face with soda lime glass as well as an example 1.

[0020]

表 4. ガラスディスクのアルカリ溶出量

単位:  $\mu\text{g}/\text{cm}^2$

	N a	L i	K	合 計
ブランク	1. 0 3 0	—	—	1. 0 3 0
比較例 2	0. 1 0 0	—	1. 6 5	1. 7 5 0
実施例 2	0. 1 0 0	—	0. 5 0	0. 6 0 0

- A blank is the processing [ in which it does not strengthen ] article of soda lime glass.

- The example 2 of a comparison is soda lime glass which carried out strengthening processing with the strengthening salt bath of 100% of potassium nitrates.

- An example 2 is soda lime glass processed by this invention approach.

As shown in Table 4, compared with that in which the soda lime glass substrate manufactured by the approach of this invention also carried out strengthening processing with non-strengthened elegance or the usual chemical-strengthening salt bath, the alkali elution volume from glass was decreasing about [ those ] to 1/2, and it was checked that chemical resistance is improving very much.

[0021]

[Effect of the Invention] As explained above, the glass substrate manufactured by carrying out chemical-strengthening processing according to the manufacture approach of the glass substrate for information record media of this invention is excellent in chemical resistance with formation of a strengthening layer on the glass substrate front face, can control remarkably the alkali migration from a glass substrate front face, and can raise the dependability of an information record medium by leaps and bounds by the ability of the glass substrate which does not have a bad influence on the information record medium formed on a glass substrate to be offered. And if the manufacture approach of this invention is followed, since chemical-strengthening down stream processing and dealkalization down stream processing will unify, the place which shortening of down stream processing is made, contributes to improvement in the productivity of the glass substrate for information record media, and therefore contributes to development of industry is very size.

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**TECHNICAL FIELD**

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[Field of the Invention] This invention relates to the information record medium which used this glass substrate for the manufacture approach of the glass substrate for information record media, and the list.

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PRIOR ART

[Description of the Prior Art] Although the substrate for information record media has been conventionally offered by the substrate made from aluminum, the substrate made from carbon, a polycarbonate substrate, etc., since surface smooth nature is excellent, the glass substrate attracts attention with the formation of amount-of-information increase in recent years. Since the surface smooth nature of a glass substrate does not have a grain boundary in glass by development of a polish technique, it is possible to reduce surface roughness to atomic level theoretically. Moreover, by the fall of the display flatness of a disk substrate, it became possible to reduce the surfacing height of the magnetic head for reading, and the recording density per unit area went up, and has led to the increment in storage capacity. Thus, although the glass disk substrate attracts attention, there is a fault of being easy to damage with the impact at the time of the handling in the production process of the glass for substrates etc., and strengthening processing by the ion exchange is usually performed.

[0003] However, it poses a problem that the rich layer of the alkali metal of a strengthening layer especially a potassium atom, or a sodium atom appears on a disk main front face in the substrate front face after chemical-strengthening processing of a glass substrate here. There is a danger that the migration of alkali-metal ion will cause reading incorrect actuation from this layer as the state of preservation of a disk and aging after subsequent information record-medium membrane formation because the carbonate and chloride of alkali metal deposit on a disk edge and the main front face. Moreover, alkali metal reacting with the record medium of a metal alloy, and causing incorrect actuation of equipment is also considered.

[0004] For this reason, after carrying out ion exchange treatment of the glass substrate front face, the alkali-metal ion of the maximum surface layer which is easy to be eluted is removed, and the dealkalization processing for raising chemical resistance and the cure against the closure of alkali-metal ion are performed. For example, there is an approach using an acid, especially strong acid like the approach of making the heated concentrated sulfuric acid contacting like the manufacture approach shown in a patent public presentation number and JP,10-226539,A. Moreover, like the manufacture approach shown in a patent public presentation number and JP,8-180402,A, into 80-100-degree C warm water, there is also a warm water art which carries out dealkalization metal ion processing of the glass substrate after ion exchange treatment by carrying out immersion processing for about 2 to 10 hours, in this case, impregnation processing of a divalent metallic ion is carried out to the maximum surface layer of the glass substrate after dealkalization metal ion processing, and the cure against the closure of alkali-metal ion is added further.

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**EFFECT OF THE INVENTION**

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[Effect of the Invention] As explained above, the glass substrate manufactured by carrying out chemical-strengthening processing according to the manufacture approach of the glass substrate for information record media of this invention is excellent in chemical resistance with formation of a strengthening layer on the glass substrate front face, can control remarkably the alkali migration from a glass substrate front face, and can raise the dependability of an information record medium by leaps and bounds by the ability of the glass substrate which does not have a bad influence on the information record medium formed on a glass substrate to be offered. And if the manufacture approach of this invention is followed, since chemical-strengthening down stream processing and dealkalization down stream processing will unify, the place which shortening of down stream processing is made, contributes to improvement in the productivity of the glass substrate for information record media, and therefore contributes to development of industry is very size.

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**TECHNICAL PROBLEM**

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[Problem(s) to be Solved by the Invention] However, since [ which needs immersion time amount at least 2 hours or more ] there are few treatment effects, impregnation processing of a divalent metallic ion is also required of the danger on the handling of using strong acid, such as heat concentrated sulfuric acid (it being heat concentrated sulfuric acid of 96% or more of concentration for example, at 100-degree-C \*\*), in these dealkalization metal ion processings, and warm water processing as after treatment. moreover, the process of [ in order for the dealkalization processing currently performed from these former to perform dealkalization processing after chemical-strengthening processing, to be carried out at a chemical strengthening and two processes of dealkalization and to avoid the drag-in to the dealkalization processing tub of a strengthening salt further ] strengthening, washing, and dealkalization processing — not constructing — it does not obtain but has also become a cost rise.

[0006] This invention makes it the technical problem to offer the glass substrate for information record media created by the effectiveness rise of workability, low-cost-izing, the chemical-strengthening art of the glass substrate for information record media which realizes the sex from Takao and a dealkalization art, and its approach by performing chemical-strengthening processing and dealkalization processing to coincidence in the manufacture approach of the glass substrate for information record media.

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MEANS

[Means for Solving the Problem] this invention person is related with the manufacture approach of the glass substrate for information record media, as a result of examining the above-mentioned technical problem wholeheartedly. The nitrate of the metal which has a bigger ionic radius smaller than potassium ion than sodium ion in strengthening salt baths, such as mixed salt of the potassium nitrate or potassium nitrate used for the chemical strengthening from the former, and a sodium nitrate, is added. By performing chemical-strengthening processing and dealkalization processing of a glass substrate to coincidence, a header and this invention were completed for the above-mentioned technical problem being attained by performing the same strengthening processing as usual.

[0008] That is, the glass substrate for information record media of this invention is carrying out predetermined time immersion of the non-strengthened glass substrate at predetermined temperature at the salt bath for chemical strengthenings, and is the creation approach of the new glass substrate for information record media of performing dealkalization to a glass substrate by one processing within the same processing tub with installation of a chemical-strengthening layer.

[0009]

[Embodiment of the Invention] Hereafter, this invention is explained to a detail.

[0010] this invention person sets to chemical-strengthening processing of a glass substrate especially paying attention to strengthening processing and dealkalization processing of the glass substrate for information record media. It is characterized by performing chemical-strengthening processing and dealkalization processing of a glass substrate by one processing within the same processing tub by adding a metal nitrate further from the former to a strengthening salt. By adding the nitrate of several% of the weight of a metal to the weight of the salt bath for strengthening, a header and this invention can be made for it being possible to perform chemical-strengthening processing and dealkalization processing to coincidence continuously.

[0011] In chemical-strengthening processing of the glass substrate for information record media, the alkali ion exchange on the front face of glass is performed at the temperature below a glass transition point, and, as for the case of one-step strengthening, in exchange of the sodium ion in glass, and the potassium ion in a strengthening salt bath, and two-step strengthening, exchange of the lithium ion in glass, sodium ion and the sodium ion in a strengthening salt bath, and potassium ion is performed. The thermal diffusion speed of the alkali-metal ion in this case is in inverse proportion to the ionic radius of each ion. Generally, under this temperature, this \*\*, and this concentration, the direction of ion with a small ionic radius has high potential energy, and can say that activity is high. Therefore, by adding the nitrate of a metal with the middle ionic radius of the ionic radius of exchangeable ion in a chemical-strengthening salt bath, the alkali ion with the small ionic radius in glass is emitted by thermal diffusion into a salt bath from a glass front face, and controls the rapid ion exchange on the front face of glass of the alkali ion which has a big ionic radius in a salt bath further. That is, the amount of alkali metal with the small ionic radius which is easy to separate after glass strengthening is decreased, and the chemical-strengthening processing by the ion exchange is attained at coincidence. That is, it is related with the glass substrate for information record media manufactured by the manufacturing method of the glass substrate for information record media using the strengthening salt bath which made it possible to perform the strengthening processing and dealkalization processing by the ion exchange on the front face of glass to coincidence, and enabled the productivity rise and the cost cut, and its strengthening salt bath, and its approach.

[0012] Moreover, by applying this invention, the productivity drive of the glass substrate for information record media and cost cut-ization can be attained. It cannot limit especially as a glass ingredient and for example, aluminosilicate system glass, soda lime glass, borosilicate glass, alumino borosilicate glass, etc. can be used as what can form a strengthening layer by chemical-strengthening processing. Moreover, limitation does not have the surface roughness of the glass substrate before performing strengthening processing, either.

[0013] However, it is required that the chemical-strengthening processing in the temperature below the glass transition point of tempered glass-ed is possible and for the nitrate of the metal added in the temperature to dissolve into a strengthening salt bath. Moreover, it is convenient, even if it is desirable that the ionic radius in the inside of the salt bath of this nitrate metal to add is within the middle of the ionic radius in each ionic state of the alkali metal in glass and a salt bath Nakakane group and it becomes large from it. In addition, 0.1 or less % of the weight of the dealkalization effectiveness is insufficient, 10% of the weight or more of the amount of dealkalization is large, and glass colors it yellow with complex ion, and is also a cost rise, and the amount which adds a metal nitrate in a strengthening salt bath does not have it. [ desirable ]

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EXAMPLE

[Example] (Example 1) The salt bath which added the 30 sections for the sodium nitrate and added the 1 section of silver nitrates for the potassium nitrate during the mixed salt bath of the 70 sections was made into the chemical-strengthening salt bath. This glass substrate ingredient was taken out from the chemical-strengthening salt bath tub after 40 minutes - 6-hour immersion using the alumino silicate glass which carried out the disk configuration whose surface roughness Ra is 0.3 micrometers, and whose diameters are 95mmphi and the thickness of 1.07mm as a glass substrate ingredient for [ strengthened ] in the chemical-strengthening salt bath maintained at the range of 360 degrees C - 450 degrees C, and the deposit salt adhering to this glass substrate after cooling was washed out with water. This was made into the strengthening sample. About the depth of the chemical-strengthening layer formed in the glass front face, it ground to the flake so that this sample could be measured with a polarization microscope, and polarization microscope observation was performed. The result was shown in Table 1.

[0015]

表 1. 処理温度と時間の変化による強化層厚みの変化

単位:  $\mu\text{m}$  (片面)

		処 理 時 間			
		40分	80分	160分	240分
処 理 時 間	360℃	30	30	40	40
	380℃	40	50	60	60
	400℃	70	90	100	100
	450℃	80	90	110	110

As shown in Table 1, it turns out that a strengthening layer (compressive-stress layer) can be introduced into a glass front face by using this invention, and the breakage in the disk breakage at the time of handling, a subsequent polish process, etc. can be mitigated sharply.

[0016] And among each sample shown in Table 1, the waterproof trial was performed about the sample processed for 40 minutes, and 380 degrees C of measurement of the alkali-metal (Na, Li, K) concentration eluted from glass were measured with atomic-absorption-analysis equipment. In addition, the waterproof trial was immersed into the Teflon (trademark) beaker which filled the glass substrate with 50ml distilled water, moved the Teflon beaker containing the glass substrate to the constant temperature bath kept at 80 degrees C, and carried out quantitative analysis of the amount of alkali metal which held for 24 hours and was eluted from the glass substrate in 80-degree C warm water with the atomic absorption method. The result was as being shown in Table 2.

[0017]

表 2. ガラスディスクのアルカリ溶出量

単位:  $\mu\text{g}/\text{cm}^2$

	Na	Li	K	合 計
ブランク	0.35	0.125	0	0.475
比較例 1	0.315	0.007	0.16	0.482
実施例 1	0.15	0.007	0.05	0.207

- A blank is the processing [ in which it does not strengthen ] article of alumino silicate glass.

The example 1 of a comparison is alumino silicate glass which carried out strengthening processing with the strengthening salt bath which mixed the potassium-nitrate 70 section and the sodium-nitrate 30 section.

- An example 1 is alumino silicate glass processed by this invention approach.

It turns out that the alkali elution volume from glass is decreasing or less [ those ] to 1/2, and chemical resistance is improving very much compared with that in which the glass substrate manufactured by the approach of this invention carried out strengthening processing with non-strengthened elegance or the usual chemical-strengthening salt bath so that clearly from Table 2.

[0018] (Example 2) Next, the salt bath which added the 1 section of silver nitrates for the potassium nitrate during the mixed salt bath of the 100 sections was made into the chemical-strengthening salt bath. About the sample which carried out chemical-strengthening processing of the soda lime glass of the same configuration as an example 1 similarly, the strengthening layer depth and an alkali elution volume were measured by this approach. Consequently, among each sample shown about the depth of a strengthening layer in Table 3 and 3, the waterproof trial was performed about the sample processed for 16 hours, and 450 degrees C of results of having measured the alkali elution volume from glass were shown in Table 4.

[0019]

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表 3. 処理温度と時間の変化による強化層厚みの変化

単位:  $\mu\text{m}$  (片面)

		処 理 時 間		
		1 時間	4 時間	1 6 時間
処 理 時 間	4 0 0 °C	1 0	2 5	5 0
	4 5 0 °C	1 0	2 5	6 0
	4 8 0 °C	1 5	3 0	7 0

As shown in Table 3, it turned out that this invention can introduce a strengthening layer (compressive-stress layer) into a glass front face with soda lime glass as well as an example 1.

[0020]

表 4. ガラスディスクのアルカリ溶出量

単位:  $\mu\text{g}/\text{cm}^2$

	N a	L i	K	合 計
ブランク	1 . 0 3 0	—	—	1 . 0 3 0
比較例 2	0 . 1 0 0	—	1 . 6 5	1 . 7 5 0
実施例 2	0 . 1 0 0	—	0 . 5 0	0 . 6 0 0

- A blank is the processing [ in which it does not strengthen ] article of soda lime glass.

- The example 2 of a comparison is soda lime glass which carried out strengthening processing with the strengthening salt bath of 100% of potassium nitrates.

- An example 2 is soda lime glass processed by this invention approach.

As shown in Table 4, compared with that in which the soda lime glass substrate manufactured by the approach of this invention also carried out strengthening processing with non-strengthened elegance or the usual chemical-strengthening salt bath, the alkali elution volume from glass was decreasing about [ those ] to 1/2, and it was checked that chemical resistance is improving very much.

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(54) 【発明の名称】 磁気記録媒体用ガラス基板の製造方法

(57) 【要約】

【課題】 情報記録媒体用ガラス基板の製造方法において、化学強化処理と脱アルカリ処理を同時に行うことにより、作業性の効率アップ、低コスト化、高生産性を実現し、その方法によって作成される情報記録媒体用ガラス基板を提供することを課題としている。

【解決手段】 ガラス基板の化学強化処理において、従来からの強化塩に更に金属の硝酸塩を添加することにより、ガラス基板の化学強化処理と脱アルカリ処理を1段階の処理で行うことを特徴とし、強化用塩浴の重量に対して数重量%の金属の硝酸塩、例えば硝酸銀を添加することにより、連続的に化学強化処理と脱アルカリ処理が同時に行うことが可能となった。

## 【特許請求の範囲】

【請求項1】情報記録媒体用ガラス基板の化学強化処理において、イオン交換による化学強化処理するため、強化塩浴に金属の硝酸塩を添加し、基板を該強化塩浴に浸漬することで化学強化と脱アルカリ処理が同時に行われることを特徴とする情報記録媒体用ガラス基板製造方法。

【請求項2】情報記録媒体用ガラス基板の化学強化処理において、イオン交換による化学強化処理するため、強化塩浴に金属の硝酸塩を添加し、基板を該強化塩浴に浸漬することで化学強化と脱アルカリ処理を同時に行うことにより製造される情報記録媒体用ガラス基板。

【請求項3】請求項1の情報記録媒体用ガラス基板の製造方法において、特にガラス中のアルカリイオンのイオン半径よりも大きく、強化塩浴中の各イオンのイオン半径よりも小さいイオン半径を持つ金属の硝酸塩を強化塩浴中に0.1～10重量%添加した強化、脱アルカリ塩浴。

【請求項4】請求項3の強化、脱アルカリ塩浴を用いて製造される情報記録媒体用ガラス基板。

【請求項5】請求項3の強化、脱アルカリ塩浴中に、特に硝酸銀を添加した強化、脱アルカリ塩浴。

## 【発明の詳細な説明】

## 【0001】

【発明の属する技術分野】本発明は情報記録媒体用ガラス基板の製造方法、並びにこのガラス基板を用いた情報記録媒体に関するものである。

## 【0002】

【従来の技術】従来、情報記録媒体用基板は、アルミ製基板やカーボン製基板及びポリカーボネート基板などにより提供されてきたが、近年情報量増大化に伴い、表面平滑性が優れることからガラス製基板が注目されている。研磨技術の発展によりガラス製基板の表面平滑性はガラスに結晶粒界がないため、理論的には原子レベルまで表面粗さを低下させることが可能である。また、ディスク基板の平坦度の低下により、読み取り用磁気ヘッドの浮上高さを低下させることが可能となり、単位面積当りの記憶密度は上昇し、記憶容量の増加に繋がっている。このようにガラス製ディスク基板は注目されているが、基板用ガラスの製造工程におけるハンドリング時の衝撃等で破損しやすいという欠点があり、通常イオン交換による強化処理が施される。

【0003】しかし、ここで問題となるのはガラス基板の化学強化処理後の基板表面に強化層のアルカリ金属、特にカリウム原子、またはナトリウム原子のリッチな層がディスク主表面に現れることである。この層からアルカリ金属イオンのマイグレーションにより、ディスクの保存状態や、その後の情報記録媒体成膜後の経時変化としてディスク端部、主表面にアルカリ金属の炭酸塩や塩化物が析出することで、読み取り誤作動を引き起こす危

険性がある。また、アルカリ金属が金属合金の記録媒体と反応して、装置の誤作動を引き起こすことも考えられる。

【0004】このため、ガラス基板表面をイオン交換処理した後、溶出し易い最表面層のアルカリ金属イオンを除去し、耐化学性を上げるための脱アルカリ処理やアルカリ金属イオンの封止対策が行われている。例えば、特許公開番号、特開平10-226539に示される製造方法のように、加熱した濃硫酸と接触させる方法のように酸、特に強酸を用いる方法がある。また特許公開番号、特開平8-180402に示される製造方法のように、80～100℃の温水中に、イオン交換処理後のガラス基板を2～10時間程度浸漬処理することで脱アルカリ金属イオン処理する温水処理方法もあり、この場合更に、脱アルカリ金属イオン処理後ガラス基板の最表面層に対して2価金属イオンの注入処理をして、アルカリ金属イオンの封止対策が加えられている。

## 【0005】

【発明が解決しようとする課題】しかし、これらの脱アルカリ金属イオン処理では、熱濃硫酸（例えば、100℃超で濃度96%以上の熱濃硫酸）等の強酸を使用することの取扱い上の危険性や、温水処理では少なくとも2時間以上の浸漬時間が必要であり、また処理効果が少ないため後処理として2価金属イオンの注入処理が必要でもある。また、これら従来から行われている脱アルカリ処理は、化学強化処理後に脱アルカリ処理を行っており、化学強化と脱アルカリの2工程で行われており、さらに、強化塩の脱アルカリ処理槽への持ち込みを避けるため強化、洗浄、脱アルカリ処理という工程を組まざるを得ず、コストアップにもなっている。

【0006】本発明は、情報記録媒体用ガラス基板の製造方法において、化学強化処理と脱アルカリ処理を同時に行うことにより、作業性の効率アップ、低コスト化、高生産性を実現する情報記録媒体用ガラス基板の化学強化処理方法と脱アルカリ処理方法、その方法によって作成される情報記録媒体用ガラス基板を提供することを課題としている。

## 【0007】

【課題を解決するための手段】本発明者は、上記の課題について鋭意検討した結果、情報記録媒体用ガラス基板の製造方法に関し、従来から化学強化に用いられている硝酸カリウムあるいは硝酸カリウムと硝酸ナトリウムの混合塩等の強化塩浴にカリウムイオンよりも小さくナトリウムイオンよりも大きなイオン半径を持つ金属の硝酸塩を添加して、従来と同様の強化処理を施すことによりガラス基板の化学強化処理と脱アルカリ処理が同時に行われることにより、上記の課題が達成されることを見出し、本発明を完成させた。

【0008】即ち、本発明の情報記録媒体用ガラス基板は、未強化のガラス基板を化学強化用の塩浴に所定温度

で所定時間浸漬することで、ガラス基板に化学強化層の導入と、脱アルカリを同じ処理槽内で1度の処理で行う新たな情報記録媒体用ガラス基板の作成方法である。

【0009】

【発明の実施の形態】以下、本発明を詳細に説明する。

【0010】本発明者は、特に情報記録媒体用ガラス基板の強化処理と脱アルカリ処理に着目し、ガラス基板の化学強化処理において、従来からの強化塩に更に金属の硝酸塩を添加することにより、ガラス基板の化学強化処理と脱アルカリ処理を同じ処理槽内で1度の処理で行うことを特徴とし、強化用塩浴の重量に対して数重量%の金属の硝酸塩を添加することにより、連続的に化学強化処理と脱アルカリ処理を同時に行うことが可能であることを見出し、本発明をなし得たものである。

【0011】情報記録媒体用ガラス基板の化学強化処理では、ガラス表面のアルカリイオン交換をガラス転移点以下の温度で行ない、1段階強化の場合はガラス中のナトリウムイオンと強化塩浴中のカリウムイオンの交換、2段階強化の場合はガラス中のリチウムイオンおよびナトリウムイオンと強化塩浴中のナトリウムイオンおよびカリウムイオンの交換が行われている。この場合のアルカリ金属イオンの熱拡散スピードは各イオンのイオン半径に反比例している。一般的に、イオン半径の小さなイオンの方が同温度、同圧、同濃度下では、ポテンシャルエネルギーが高く、活性が高いと言える。そのため、交換イオンのイオン半径の中間のイオン半径を持つ金属の硝酸塩を化学強化塩浴中に添加することにより、ガラス中のイオン半径の小さなアルカリイオンは、熱拡散によりガラス表面から塩浴中に放出され、更に塩浴中の大きなイオン半径を持つアルカリイオンのガラス表面への急激なイオン交換を抑制する。つまり、ガラス強化後に遊離しやすい小さなイオン半径を持つアルカリ金属量を減少させ、同時にイオン交換による化学強化処理が可能となる。即ち、ガラス表面のイオン交換による強化処理と脱アルカリ処理とを同時に行うことを可能にして、生産性アップとコストダウンを可能にした強化塩浴と、その強化塩浴を用いた情報記録媒体用ガラス基板の製造法、\*

\*またその方法で製造される情報記録媒体用ガラス基板に関するものである。

【0012】また本発明を適用することにより、情報記録媒体用ガラス基板の生産性向上とコストダウンを図ることができる。ガラス材料としては特に限定するものでなく、化学強化処理で強化層を形成することのできるものとして、例えばアルミノシリケート系ガラス、ソーダライムガラス、ホウケイ酸ガラス、アルミノホウケイ酸ガラス等を用いることができる。また、強化処理を行う前のガラス基板の表面粗さも限定はない。

【0013】ただし、被強化ガラスのガラス転移点以下の温度における化学強化処理が可能であること、またその温度において添加する金属の硝酸塩が強化塩浴中に溶解することが必要である。また、この添加する硝酸塩金属の塩浴中でのイオン半径がガラス中のアルカリ金属と塩浴中金属の各イオン状態におけるイオン半径の中間内にあることが好ましく、それより大きくなっても支障はない。なお、金属の硝酸塩を強化塩浴中に添加する量は、0.1重量%以下では脱アルカリ効果が不十分であり、10重量%以上は脱アルカリ量が大きく、またガラスが銀イオンにより黄色に着色し、コストアップでもあり好ましくない。

【0014】

【実施例】(実施例1)硝酸ナトリウムを30部、硝酸カリウムを70部の混合塩浴中に硝酸銀を1部添加した塩浴を化学強化塩浴とした。被強化用ガラス基板材料として、表面粗さRaが0.3μm、直径が95mmφ、厚み1.07mmのディスク形状をしたアルミノシリケートガラスを用い、該ガラス基板材料を360℃~450℃の範囲に保たれた化学強化塩浴中に40分~6時間浸漬後、化学強化塩浴槽から取りだし、冷却後該ガラス基板に付着している析出塩を水で洗い落とした。これを強化サンプルとした。ガラス表面に形成された化学強化層の深さについて、このサンプルを偏光顕微鏡で測定できるように薄片に研磨して、偏光顕微鏡観察を行なった。その結果を表1に示した。

【0015】

表1. 処理温度と時間の変化による強化層厚みの変化  
単位: μm (片面)

		処 理 時 間			
		40分	80分	160分	240分
処理時間	360℃	30	30	40	40
	380℃	40	50	60	60
	400℃	70	90	100	100
	450℃	80	90	110	110

表1に示したように、本発明を利用することでガラス表面に強化層(圧縮応力層)が導入できることが分かり、ハンドリング時のディスク破損や、その後の研磨工程などにおける破損を大幅に軽減できる。

【0016】そして、表1で示した各サンプルの内、380℃、40分処理したサンプルについて耐水性試験を

行い、ガラスから溶出したアルカリ金属(Na、Li、K)濃度の測定を原子吸光分析装置にて測定した。なお耐水性試験は、ガラス基板を50mlの蒸留水で満たしたテフロン(登録商標)ビーカー中に浸漬し、そのガラス基板の入ったテフロンビーカーを80℃に保った恒温水槽に移し、80℃の温水中に24時間保持してガラス

基板から溶出したアルカリ金属量を原子吸光法で定量分 \* 【0017】  
析した。その結果は表2に示す通りであった。 \*

表2. ガラスディスクのアルカリ溶出量

単位:  $\mu\text{g}/\text{cm}^2$

	Na	Li	K	合計
ブランク	0.35	0.125	0	0.475
比較例1	0.315	0.007	0.16	0.482
実施例1	0.15	0.007	0.05	0.207

・ブランクはアルミノシリケートガラスの未強化処理品。

比較例1は硝酸カリウム70部、硝酸ナトリウム30部を混合した強化塩浴で強化処理したアルミノシリケートガラス。

・実施例1は本発明方法で処理したアルミノシリケートガラス。

表2から明らかなように、本発明の方法で製造したガラス基板は、未強化品あるいは通常の化学強化塩浴で強化処理したものとは比べ、ガラスからのアルカリ溶出量はそれらの1/2以下に減少しており、耐化学性が非常に向上していることが分かる。

【0018】(実施例2)次に、硝酸カリウムを100部の混合塩浴中に硝酸銀を1部添加した塩浴を化学強化塩浴とした。実施例1と同じ形状のソーダライムガラスを同様に化学強化処理したサンプルについて、強化層深さとアルカリ溶出量を同方法で測定した。その結果、強※

表4. ガラスディスクのアルカリ溶出量

単位:  $\mu\text{g}/\text{cm}^2$

	Na	Li	K	合計
ブランク	1.030	—	—	1.030
比較例2	0.100	—	1.65	1.750
実施例2	0.100	—	0.50	0.600

・ブランクはソーダライムガラスの未強化処理品。

・比較例2は硝酸カリウム100%の強化塩浴で強化処理したソーダライムガラス。

・実施例2は本発明方法で処理したソーダライムガラス。

表4に示したように、本発明の方法で製造したソーダライムガラス基板も、未強化品あるいは通常の化学強化塩浴で強化処理したものとは比べ、ガラスからのアルカリ溶出量はそれらの1/2程度に減少しており、耐化学性が非常に向上していることが確認された。

【0021】

【発明の効果】以上説明したように、本発明の情報記録

※化層の深さについては表3に、そして表3で示した各サンプルの内、450℃、16時間処理したサンプルについて耐水性試験を行い、ガラスからのアルカリ溶出量を測定した結果を表4に示した。

【0019】

表3. 処理温度と時間の変化による強化層厚みの変化

単位:  $\mu\text{m}$  (片面)

		処 理 時 間		
		1時間	4時間	16時間
処理時間	400℃	10	25	50
	450℃	10	25	60
	480℃	15	30	70

表3に示したように、本発明はソーダライムガラスでも、実施例1と同様にガラス表面に強化層(圧縮応力層)が導入できることが分かった。

【0020】

媒体用ガラス基板の製造方法に従って化学強化処理をして製造されたガラス基板は、ガラス基板表面に強化層の形成とともに耐化学性に優れており、ガラス基板表面からのアルカリマイグレーションを著しく抑制し、ガラス基板上に成膜される情報記録媒体に悪影響を及ぼさないガラス基板を提供できることで、情報記録媒体の信頼性を飛躍的に向上させることができるものである。そして、本発明の製造方法に従えば、化学強化処理工程と脱アルカリ処理工程とが一体化しているため、処理工程の短縮化ができ、情報記録媒体用ガラス基板の生産性の向上に寄与し、よって産業の発展に寄与するところは非常に大であります。